

SYNTHESIS AND STRUCTURAL STUDY OF NICKEL (II)

BAKELITE NANOCOMPOSITE BY X-RAY DIFFRACTION

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ABSTRACT

The Nickel (II) Bakelite composite has been synthesized using phenol formaldehyde by chemical precipitation method. To obtain the nickel nanoparticles synthesized phenol formaldehyde decomposed at 800°C. The composite so had attributes of both metal and polymer and their properties depend on proportion and compatibility of various components of composite. The chemical composition and crystallographic structure of Ni nanoparticle is confirmed by XRD measurement. The XRD analysis reveals the size of the nanoparticle is 24 nm, and the crystal structure is monoclinic.

KEYWORDS: Phenol, Formaldehyde, XRD, Bakelite composite. Nickel Chloride tetra hydrated

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INTRODUCTION

The progress in nanocomposites is varied and covered many industries. Nanocomposites can be made with a variety of enhanced thermal, physical and other properties. They can be synthesized using simple and inexpensive techniques. Conducting polymers based nanocomposites have electrical and electrochemical properties that show great promise application in transistor, batteries, and light emitting diodes.¹

Nanoparticles into a polymeric system may versatile magnetic, semiconducting, Mechanical and optical properties. Magnetic nanocomposite possesses giant magneto resistive effect.²⁻³ Hence have potential application in super high density data storage devices.⁴⁻⁶ Thermal stability of nanocomposite has great application in gas sensors, biosensors, nanogenerator and photo detectors etc⁷

High density crossed link thermosetting polymer widely used in plastic, varnish, laminates, lacquers, and sports equipments. They have polycondensation of phenol and formaldehyde under acidic or basic condition resulting formation of resole and nevolak. In basic condition resole form and acidic condition nevolaks are formed. Phenolic resin has good thermos-oxidative resistance, extensive research been done to improve thermal properties, tensile strength through modification of their structure with introduction of some metal ion to produce metal matrix composite MMC. MMCs are playing major roll in various sectors. i.e. transportation, electronics.⁸

Through this paper simple synthesis of Cr (III) Bakelite nanocomposite has been reported and further composite is characterized by XRD to study the structural attribute. This work is an attempt such as material that

has versatile electrical and magnetic properties and can be used as high performance material. These improve the properties of bakelite useful for industrial application.

EXPERIMENTAL

Materials

Important chemicals like Phenol C_6H_5OH (Central Drug House Pvt. Ltd.), Formaldehyde $HCHO$ (Central Drug House Pvt. Ltd), CH_3COOH Glacial Acetic Acid (Fisher Scientific), HCl Hydrochloric Acid (Fisher Scientific), and metal salt nickel chloride tetra hydrated $NiCl_2 \cdot 4H_2O$ (Central Drug House Pvt. Ltd.) Distilled water which was used to prepare composites was chemically pure grade. Metal solution was prepared by dissolving appropriate amount of its chloride salt in distilled water.

Synthesis

Synthesis of Nanocomposite takes place in two steps-

- **Bakelite Chromium Metal Composite Complex**

To prepare 1 normal Ni^{+2} metal salt solution prepared in distilled water. Then in a small beaker 6gm phenol, 12 ml formaldehyde, then added in 15 ml glacial acetic acid continues stirring. Then addition of (30- 35ml app.) concerted hydrochloric acid and 20 ml of 1N solution added in it. The complex intermixed solution is then heating is take place for about thirty minute by heating plate. After 30 minute a large light pink luster was formed. The solid sample is been purified by the washing with distilled water. The excess of metal ion and the impurities present on the sample is purified by the washing. The polymers composite with variable concentration of inorganic salt were formed. Then it kept in to the desiccators for the drying for fifteen days.

- **Synthesis of Nanocomposite by Decomposition of Bakelite Composite Complex**

In order to get the bakelite nanocomposite decomposition of bakelite chromium metal complex is take place at $800^{\circ}C$. The complex decomposition takes place at 45 minute.

NANOCOMPOSITE CHARACTERIZATION

Powder X-Ray Diffraction

After preparing Bakelite composite of variable nickel chloride salt concentration, the composites were crushed and subjected to powder XRD analysis. X-Ray technique give information about the crystal system, crystal structure, chemical composition and physical properties of material. The Powder diffraction method is suited well for identification and characterization of polycrystalline phases.

Powder XRD X- Ray diffraction studies were performed on the an X-ray diffractometer(ICDD Grant- in-Aid) Under ambient condition using $Cu K\alpha$ ($1.54051^{\circ} A$) radiation, Beta- filter and scintillation counter as detector at 40 kv and 40 mA on rotation between 20° to 80° at 2θ scale at 7 sec. The sample was made on " $NiCO_3$ " and toluenesulfonic acid monohydrate in stoichiometric amounts at room temperature and evaporating the resulting solution at 30 C. Temperature of Data Collection: Pattern taken at 295 K. Data collection flag: Ambient. Stober,

RESULTS AND DISCUSSIONS

XRD pattern of pure Bakelite nanocomposite has maximum intensity diffraction peak at $2\theta = 43.5^\circ$ with d spacing 12.50290 Å which indicate the presence of crystalline structure and the crystal system is orthorhombic. X-ray diffraction patterns of Ni Bakelite composites show in figure 1.

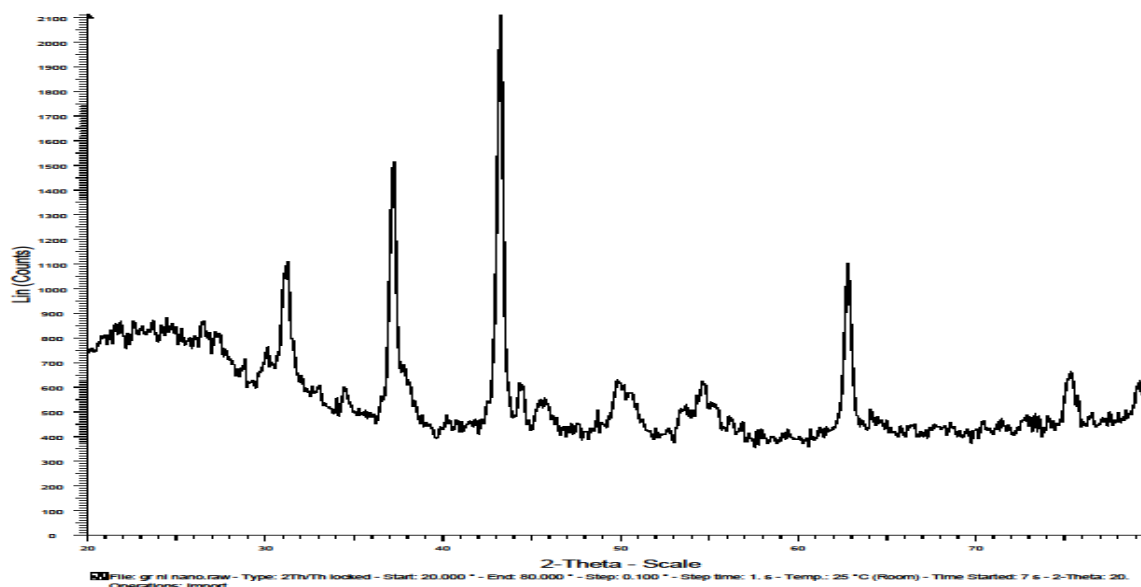


Figure 1: XRD Pattern of $\text{NiCl}_2 \cdot 4\text{H}_2\text{O}$ Nickel Bakelite Nano Composite

CONCLUSIONS

There is compatibility between various components of salt and polymer thus synthesis of Ni composite is possible. The XRD analysis proves the Nano structure of the Ni particle through its crystal analysis and spacing. Crystallinity of composite decrease with addition of salt.

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